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Article

Effects of sodium carbonate and *Sapindus laurifolia* in combination on the degumming of Muga silk fibers

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Abstract: Muga silk is the most important composite material used in textile manufacturing in India. Muga silk is derived from the Muga silkworms, namely *Antheraea assamensis* Helfer. The golden yellow silk yarn is the fanciest because it has strange properties like being able to handle different textures well, being bright, and lasting a long time. Fibrin (a fibrous protein) and sericin (a globular protein) are the two most important protein units that make up silk. To make silk usable in the textile business, sericin, a gum, has to be cleaned off the surface of the silk. Generally, surface active agents are used in the extraction of sericin from silk material. The present research describes a comparison between the degumming activity of a natural surfactant saponin isolated from *Sapindus laurifolia* and *Sapindus laurifolia*-Na_2CO_3 mixed system. The effect of the salt Na_2CO_3 on the degumming ability of *Sapindus laurifolia* is systematically studied and reported. The surface morphology of the raw and degummed silk fibers is compared using scanning electron microscope.

Keywords: fibroin, sapindus laurifolia, sericin, saponin, and Na_2CO_3

1. Introduction

Muga silk, derived from the muga silkworm species known as *Antheraea assamensis*, is a key component in textile manufacturing in India. These silkworms eat som (*Machilus bombycina*) and sualu (*Litsaea polyantha*) leaves. The golden yellow silk yarn is the costliest because of its distinct features, including superior texture, luster, and durability. Sericin is a natural gum-like proteinaceous material secreted by the silkworms alongside the fibroin, which forms the silk fibers. Degumming of silk cocoons is required to remove the sericin for improving silk quality, enhancing dyeing ability, and increasing the strength and elasticity of the fiber. Sericin is soluble in hot water that contains alkali, detergents, enzymes, or organic acids, but fibroin is insoluble in water due to its highly organized crystalline fibrous structure. Fibroin has long been employed in the production of clothing and home textiles. Scaffolds composed of different combinations of silk fibroin have been utilized in tissue design [1], drug delivery, and regenerative medicine [2]. On the other hand, sericin possesses notable medical attributes such as antimicrobial, anticoagulant, and antioxidant capabilities and is employed in tissue engineering applications. There are several reagents that can be employed for degumming silk fibroin, each with its own advantages and disadvantages. Some common reagents employed in degumming process are soap, alkaline solution [3], enzyme solution [4] and acid solution [5]. The soap dissolve and remove the sericin from the silk fibers However, this method may require longer treatment times and multiple rinses to ensure complete degumming. Excessive dosages of alkaline reagents can damage the silk fibers if not controlled properly. However, enzymes such as proteases or serine proteases can selectively degrade sericin without affecting the silk fibroin.

In the recent years some saponin based natural surfactant isolated from fruits of *Sapindus laurifolia* [6] and *Acaia concinna*plant [7] we're being applied for extraction of sericin from cocoon surface. Saponins can effectively degum silk due to their ability to emulsify fats and proteins. When saponins are applied to silk fibers, they help to break down and dissolve the sericin, allowing it to be easily washed away. This results in silk fibers that are smoother, softer, and more lustrous. The use of saponins as a degumming agent for silk is advantageous because they are natural and environmentally friendly. Additionally, they are relatively gentle compared to harsher chemical degumming agents, which can potentially damage the delicate silk fibers. Butthe effect of salt on the surface activity and degumming activity of saponin is not yet studied. Here is an attempt to explore the synergetic effect of sodium carbonate (Na_2CO_3) on the degumming tendency of saponin isolated from the plant *S. laurifolia* has been initiated.

2. Material and methods

2.1. Collection of raw silk fiber and S.laurifolia fruit

Raw silk strands were purchased from the Guwahati, India, city market. Fruit of the *S. laurifolia* plant was collected from the Koraput, Odisha, India, and used for extraction of saponin for degumming purpose. Na_2CO_3 was procured from Merck India Limited. Demineralized water was used in the laboratory to perform the degumming procedure.

2.2. Extraction of Saponin solution from Sapindus laurifolia fruit

The pericarps of approximately 100 grams of *S. laurifolia* fruit were removed from the seeds. The pericarps sample was subsequently pulverized into a powdered form and subsequently mixed with 1000 mL of water using a magnetic stirrer for 2 hours. The supernatant liquid was obtained by centrifuging the aqueous froth at 400 rpm for approximately 45 minutes following the completion of the stirring procedure. The degumming experiment was conducted using the supernatant liquid.

2.3. Degumming of raw silk

10 gm of Muga silk sample was dipped in 200 ml of aqueous saponin sample and stirred with a magnetic stirrer for 1 hour at 100 $^\circ C$. Following the process, the silk specimen was washed several times with tap water before being dried and weighed. Weight loss (%) was measured. The process was repeated three times.

$$Degumming\% = \frac{(\mathrm{W1} - \mathrm{W2})}{\mathrm{W1}} \times 100,$$

where W_1 is the primary weight of silk individual (before degumming process) and W_2 is the ultimate weight of silk individual (after degumming process).

2.4. Scanning electron microscope image

We investigated the surface morphological properties of silk fibers and degummed silk fibers using scanning electron microscopy (Carl ZEISS Evo 18, SEM). Silk fibroin fibers are irregularly cylindrical in shape, even though a single silk fibroin thread has a diameter of 12 micrometers. After coating the silk specimens with gold, they were examined with an excited electron beam at 10 kilovolts.

3. Results and discussion

3.1. Optimization of S. laurifolia-Na_2CO_3 mixed system for degumming

The concentration of a degumming agent significantly impacts the degumming ratio, which refers to the efficiency with which the agent removes gums (sericin) from cocoon surface. Higher concentration of the degumming agent generally leads to a more efficient removal of gums, resulting in a higher degumming ratio [8]. This is because more active surfactant molecules are available to interact with and break down the gum. There is typically an optimal concentration beyond which no significant increase in the degumming is observed. This is because the degumming process reaches a point of saturation where additional degumming agent does not lead to more gums being removed. The Critical Micellar Concentration (CMC) plays a significant role in the degumming process, particularly when surfactants are used as degumming agents. The CMC is the concentration of surfactants in a solution at which micelles start to form. Below this concentration, surfactants exist primarily as individual molecules. Above this concentration, surfactants aggregate to form micelles, which are spherical structures with hydrophobic tails inward and hydrophilic heads outward [9–11].This micellar structure is responsible solubilising the sericin of untreated silk. When the concentration of the surfactant is below the CMC, there are not enough surfactant molecules to form micelles. This results in lower efficiency in solubilizing and removing impurities like sericin from silk surface. At CMC, micelles form, significantly enhancing the ability of the surfactant to solubilize hydrophobic impurities in its micellar core. This increases the degumming ratio, as micelles can effectively encapsulate and remove these impurities.

Figure 1 describes the variation of percentage of degumming with saponin concentration. With and without the addition of Na_2CO_3.Concentration of saponin isolatedfrom *S. laurifolia* was applied in the range of 0.001 to 0.020 g/cc. It was demonstrated that, the percentage of degumming increased with an increase in the saponin concentration. The results plotted in the graph (Figure 1) showed that maximum degumming of Muga silk occurred at 0.018 g/mL of saponin which is just above the CMC of surfactant [9].With further increase in the concentration of saponin no significant increase in the degumming was observed. The CMC of a surfactant is a critical parameter in the degumming process. Maintaining the surfactant concentration at or just above the CMC of saponin (0.018g/mL) degumming ratio can be optimized by efficiently removing impurities. But with the addition of salt, Na_2CO_3 in the concentration range 0.0001 to 0.0005 g per mLthe percentage of degumming increases. But the optimized concentration of salt is 0.0003 g per mL. Above this concentration of Na_2CO_3 there is no further increase in the percentage of degumming. Adding salt causes CMC to drop by lowering the repulsive action between the charged head groups of surfactant molecules, hence preventing micelle formation. Reducing the repulsions by adding salt ions helps micelle to develop at a smaller surface-active agent concentration. This phenomenon of decreasing the CMC has a significant role in the increase of percentage of degumming by decreasing the amount of saponin to large extent and by adding a very little amount of Na_2CO_3.

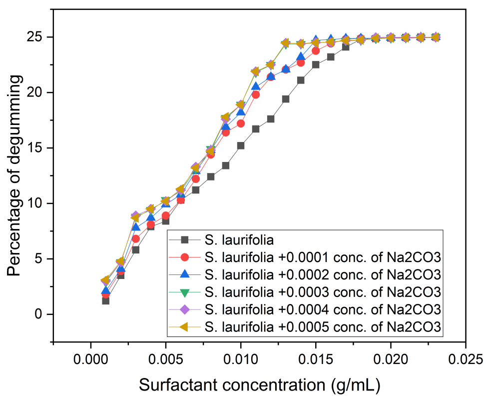

Figure 1. Variation of Surfactant Concentration on degumming percentage

This approach of decreasing the CMC balances effective degumming with economic and environmental considerations, preserving the quality of the fibers being processed.

3.2. Surface morphology analysis

To get useful insights into the structure and characteristics of silk fibers at a microscopic level, it is possible to study the surface morphology of silk fibers using a scanning electron microscope (SEM). Image obtained from a SEM of a silk fiber can provide specific information regarding a number of features of the fiber's properties and characteristics. Surface morphology is a piece of knowledge that is very essential. The surface texture of silk threads can also be revealed by SEM images. This includes the smoothness, roughness, and any surface abnormalities. By delivering images with a high resolution and in three dimensions, SEM makes it possible to conduct an exhaustive investigation into the structural and physical characteristics of silk fibers, which is beneficial for both research and industrial applications. SEM image of saponin-Na_2CO_3 degumming is depicted in Figure 2 representing the effective degumming of both the system.

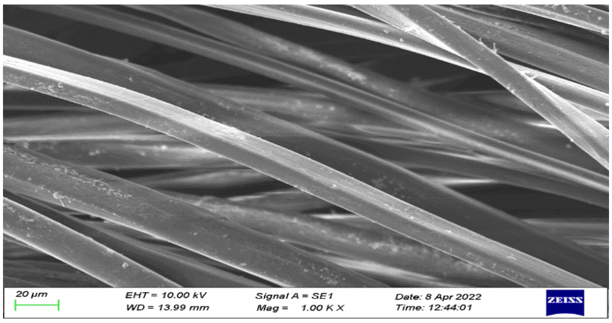

Figure 2. SEM image of saponin degumming and saponin-Na_2CO_3 degumming

4. Conclusion

The present work is a comparison between the degumming capability of saponin extracted from *Sapindus laurifolia* and saponin- Na_2CO_3system. In case of saponin alone roughly 24.9% degumming occurs at the concentration of 0.018g per mLwhich is CMC of the surfactant. However, the addition of little amount of sodium carbonate (0.0001 to 0.0005g per mL) significantly increases the degumming percentage, simultaneously decreasing amount of saponin in the degumming solution. The maximum concentration of $Na_2CO_3$is the 0.0003g per mL. The mixed system saponin- Na_2CO_3 able to successfully degum silk fibers while maintaining their grade and potency, as evidenced by the micrograph, which displayed very fine longitudinal striations of silk fibers.

Data availability statement: The authors declare that the data presented in the study confirms the conclusions. The article contains the necessary information, and the raw data that supports the results is accessible upon request from the authors. The published publication encompasses all graphics and data acquired or generated throughout the project.

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